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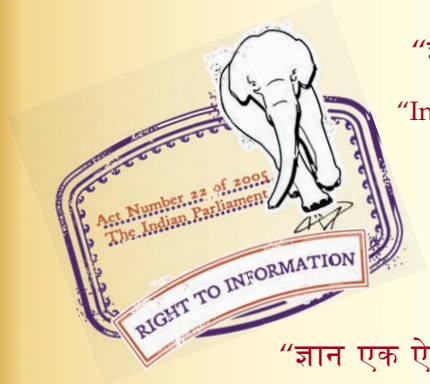

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METHOD FOR DETERMINATION OF CHLORIDE CONTENT OF TEXTILE MATERIALS

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METHOD FOR DETERMINATION OF CHLORIDE CONTENT OF TEXTILE MATERIALS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 31 July 1967, after the draft finalized by the Textile Chemistry Sectional Committee had been approved by the Textile Division Council.

0.2 In textile industry, textile materials undergo various treatments in course of which extraneous matter of various types, such as sizing or finishing material, water-soluble salts (chlorides and sulphates) is gathered by or added to the textile materials. Such water-soluble substances, if present, in more than certain quantities may have deleterious effects on the fibrous material or on other materials with which they are associated in use and may, therefore, affect their performance in service. It is hoped that this standard will be useful for determining the chloride content in aqueous extract of textile materials.

0.3 The gravimetric and volumetric methods for estimating the chloride content in textile materials are prescribed in this standard. The potentiometric titration method which is suitable for very small quantities of chloride present in textile materials is also given as third method.

0.4 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

---

1. SCOPE

1.1 This standard prescribes the methods for determination of water-soluble chloride in textile materials and the procedure for extracting the textile materials with water.

2. PRINCIPLE

2.1 The aqueous extract of textile material is prepared, the chloride content is determined, either gravimetrically or volumetrically and expressed as the percentage of the weight of the conditioned material.

*Rules for rounding off numerical values (*revised*).

3. SAMPLING

3.1 Sampling for Fibre and Yarn

3.1.1 *Lot* (*Fibre or Yarn*) — The quantity of fibre or yarn from the same source shall constitute a lot. If the lot contains more than 200 kg of fibre or yarn, it shall be divided in sub-lots each weighing 200 kg or less.

3.1.2 From a sub-lot 15 increments each approximately weighing 10 g shall be taken from different parts so that a representative sample is obtained. All the increments thus collected shall be thoroughly mixed. This shall constitute the **test sample.**

3.2 Sampling for Fabrics

3.2.1 *Lot* (*Fabric*) — The quantity of fabrics manufactured under relatively uniform conditions shall constitute a lot.

3.2.2 The number of pieces to be selected from a lot shall be as given below. The pieces thus selected shall constitute the gross sample:

Lot Size	*Sample Size*
Up to 100	3
101 ,, 300	4
301 ,, 500	5
501 and above	7

3.2.3 From each piece in the gross sample about 25 g of fabric shall be taken out from at least two different parts. The parts shall then be cut into further smaller pieces and thoroughly mixed. The pieces thus collected shall constitute the **test sample.**

4. TEST SPECIMENS

4.1 From the test sample, draw at least two test specimens each weighing about 10 g. Cut the test specimens into small pieces. If the sample under analysis is loose fibre, take about 5 g of the test specimen.

5. CONDITIONING OF TEST SPECIMENS

5.1 Prior to test, the test specimens shall be conditioned for 24 hours to moisture equilibrium in a standard atmosphere at 65 $\pm$ 2 percent relative humidity and 27° $\pm$ 2°C temperature (*see also* IS : 196-1966*).

6. APPARATUS

6.1 Flat-Bottom Flask — of a suitable capacity with a glass stopper.

6.2 Water-Cooled Condensers

6.3 Sintered-Glass Crucible — porosity 4.

*Atmospheric conditions for testing (*revised*).

6.4 Titration Vessel and Reference Half-Cell — With suitable *p*H meter as used with glass electrode balanced to read in millivolts, or galvanometer and tapping key. A convenient arrangement is shown in Fig. 1. The half-cell is filled with a suspension made by dissolving 14 g of sodium oxalate and 10 g of potassium nitrate in one litre of distilled water, adding with constant stirring, 100 ml of 0·1 N silver nitrate solution.

NOTE — The stock of suspension should be kept in dark glass bottle.

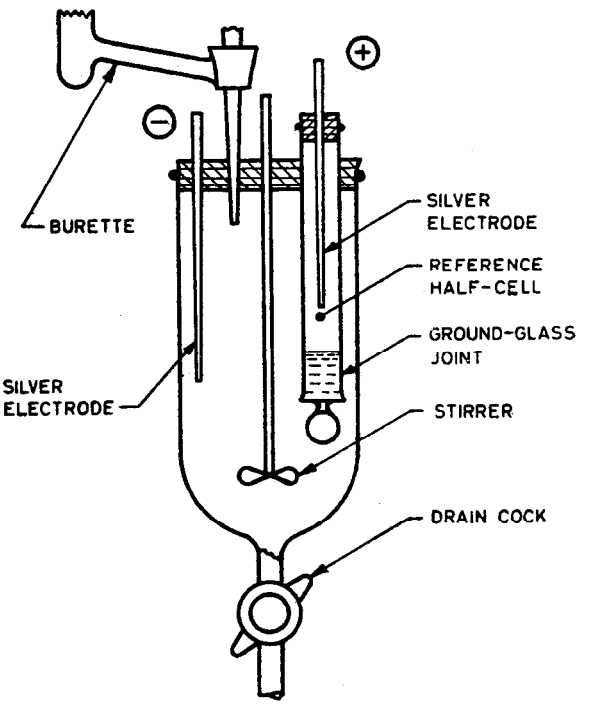

FIG. 1 TITRATION VESSEL AND REFERENCE HALF-CELL

7. QUALITY OF REAGENTS

7.1 Unless specified otherwise pure chemicals shall be employed in tests and distilled water (*see* IS : 1070-1960*) shall be used where the use of water as reagent is intended.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the test results.

*Specification for water, distilled quality (*revised*).

8. PREPARATION OF AQUEOUS EXTRACT

8.1 Condition the test specimens to moisture equilibrium in the standard atmosphere and weigh accurately each test specimen.

8.2 Put a test specimen in the flask and add sufficient amount of water to it to make liquor in material ratio of 20 : 1 (*see* Note). Connect the flask to the condenser and bring rapidly to the boil and continue to boil the liquor gently for 60 minutes. Disconnect and remove the flask while the liquor is still boiling and close it immediately with the glass stopper fitted with the stopcock. Rapidly cool the flask to room temperature (27°C). Do not remove or open the tap until ready for filtration.

NOTE — If the test specimen is wool in any form, felt or loose fibre masses of any composition, the liquor to material ratio should be 50 : 1.

8.3 Similarly prepare separate extracts for each of the remaining test specimens.

9. FIRST METHOD (GRAVIMETRIC)

9.1 Reagents

9.1.1 *Silver Nitrate Solution* — 0·1 N.

9.1.2 *Nitric Acid*

a) concentrated (sp gr 1·42)

b) 0·5 percent (*w/v*)

9.2 Procedure

9.2.1 Take a suitable measured portion of extract (*see* **8.2**). Add 5 ml of concentrated nitric acid per 100 ml, boil for 5 minutes and leave it overnight. Filter through a paper-pulp pad, wash with distilled water and add a slight excess of 0·1 N silver nitrate solution to the combined filtrate and washings. Heat the solution, protected from direct light, in a water-bath until the precipitate is coagulated and supernatant liquor is clear. Verify completeness of precipitation by adding a drop of 0·1 N silver nitrate solution to the supernatant liquor. Allow it to cool overnight in the dark and then filter through a tared sintered-glass crucible. Wash the precipitate with 0·5 percent nitric acid until the washings give no opalescence when tested with sodium chloride solution. Dry the crucible first at 100°C and finally to constant weight at 130° to 150°C.

9.2.2 Carry out a blank determination.

9.2.3 Calculate the chloride content of the test specimen by either of the following formulae:

a) For materials in yarn and fabric form other than wool (*see* Note 1):

$$P = \frac{495 \times (A - B)}{V}$$

b) For wool in any textile form and for felts and loose fibre masses of any composition (*see* Note 2):

$$P = \frac{1\,237 \times (A - B)}{V}$$

where

P = percentage, by weight, of water-soluble chloride as chloride ion;

A = weight, in g, of the precipitate obtained in the test (*see* **9.2.1**);

B = weight, in g, of the precipitate obtained in blank (*see* **9.2.2**); and

V = volume, in ml, of the extract taken for the test (*see* **9.2.1**).

NOTE 1 — 100 ml of extract are equivalent to 5·0 g of conditioned test specimen.

NOTE 2 — 100 ml of extract are equivalent to 2·0 g of conditioned test specimen.

9.2.4 Repeat the test with the extracts of the remaining test specimens.

9.2.5 Calculate the average of the values obtained as in **9.2.3** and **9.2.4**.

10. SECOND METHOD (VOLUMETRIC)

10.1 Reagents

10.1.1 *Standard Silver Nitrate Solution* — 0·1 N.

10.1.2 *Standard Potassium Thiocyanate Solution* — 0·1 N.

10.1.3 *Ferric Alum Indicator Solution* — Dissolve 100 g of ferric ammonium sulphate in 250 ml of water. Heat the solution to boiling and add concentrated nitric acid (sp gr 1·42) slowly until the red colour disappears.

NOTE — The amount of nitric acid used should be between 6 and 15 ml.

10.2 Procedure

10.2.1 Take a suitable measured portion of extract. Acidify it with 5 ml of nitric acid. Add 5 ml of 0·1 N silver nitrate solution and 5 ml

ferric alum indicator solution. Add sufficient amount of nitrobenzene (*see* Note). Titrate the excess of the silver nitrate against 0·1 N potassium thiocyanate solution till the first appearance of faint pink colour.

NOTE — About 1 ml of nitrobenzene is required in every 0·05 g of chloride.

10.2.2 Calculate the chloride content of the test specimen by either of the following formulae:

a) For materials in yarn and fabric form other than wool (*see* Note 1):

$$P = \frac{(AB - CD) \times 3{\cdot}55}{V} \times 20$$

b) For wool in any textile form and for felts and loose fibre masses of any composition (*see* Note 2):

$$P = \frac{(AB - CD) \times 3{\cdot}55}{V} \times 50$$

where

P = percentage, by weight, of water-soluble chloride as chloride ion;

A = volume, in ml, of silver nitrate solution;

B = normality of silver nitrate solution;

C = volume, in ml, of potassium thiocyanate, required for back titration;

D = normality of potassium thiocyanate solution; and

V = volume, in ml, of extract taken for test.

NOTE 1 — 100 ml of extract is equivalent to 5·0 g of conditioned test specimen.

NOTE 2 — 100 ml of extract is equivalent to 2·0 g of conditioned test specimen.

10.2.3 Repeat the test with the extracts of the remaining test specimens.

10.2.4 Calculate the average of the results obtained as in **10.2.2** and **10.2.3.**

11. THIRD METHOD (POTENTIOMETRIC TITRATION)

11.1 Reagents

11.1.1 *Silver Nitrate Solution* — 0·01 N.

11.1.2 *Nitric Acid* — concentrated (sp gr 1·42).

11.2 Procedure

11.2.1 Take a suitable measured portion of the extract (*see* **8.2**). Add 5 ml of concentrated nitric acid per 100 ml of extract and boil for five

minutes. Cool rapidly to room temperature and transfer to the titration vessel. Start the stirrer, connect the electrodes to the *p*H meter balanced to read in millivolts and titrate with 0·01 N silver nitrate solution until the galvanometer first indicates zero.

11.2.2 Carry out a blank determination.

NOTE — Before each test, it should be verified that all parts of the apparatus are clean. The silver electrodes should be cleaned with very fine abrasive or a suitable chemical method. Discard and replace the silver oxalate suspension at the first sign of darkening, and to delay darkening half-cell should be shielded from light when not in use. The suspension bottle should be shaken thoroughly before replenishing the half-cell. Take particular care that electrolytes do not come into contact with the junction between the silver wire electrodes and their leads to the *p*H meter. Flush the electrolyte junction after each determination by easing the stopper at the bottom of the half-cell to allow fresh extract suspension to flow into the junction.

11.2.3 Calculate the chloride content of the test specimen by either of the following formulae:

a) For materials in yarn and fabric form, other than wool (*see* Note 1):

$$P = \frac{0{\cdot}71 \times (V_1 - V_2)}{V}$$

b) For wool in any textile form and for felts and loose fibre masses of any composition (*see* Note 2):

$$P = \frac{1{\cdot}77 \times (V_1 - V_2)}{V}$$

where

P = percentage, by weight, of chloride content as chloride ion;

V_1 = volume, in ml, of 0·01 N silver nitrate solution required for test (*see* **11.2.1**);

V_2 = volume, in ml, of 0·01 N silver nitrate solution required for blank (*see* **11.2.2**); and

V = volume, in ml, of extract taken for the test.

NOTE 1 — 100 ml of the extract are equivalent to 5·0 g of conditioned test specimen.

NOTE 2 — 100 ml of the extract are equivalent to 2·0 g of conditioned test specimen.

11.2.4 Repeat the test with the extracts of the remaining test specimens and calculate the percentage of water-soluble chloride in each test specimen.

11.2.5 Calculate the average of the values obtained as in **11.2.3** and **11.2.4**.

12. REPORT

12.1 Report the value obtained as in **9.2.5**, **10.2.4** or **11.2.5** as the percentage of water-soluble chloride as chloride ions in textile materials.

NOTE — If the percentage of the water-soluble chlorides is to be expressed as sodium chloride, then multiply P by 1·65.

12.2 Report also the method used (whether gravimetric, volumetric or potentiometric titration).

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